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Investigation of the corrosion resistance of cold-rolled low-carbon steel in an acidic medium

Olushola Bamidele Nenuwa*, Olugbenga Olabode Oke

ABSTRACT

The corrosion resistance of cold-rolled low-carbon steel in a hydrochloric acid medium was investigated. Low-carbon steel samples were cold-rolled at a percentage reduction range of 10% to 50% in increments of 10%. Some of the cold-worked samples were annealed at 700°C, held there for one hour and cooled in the furnace to room temperature. A corrosion test was carried out on the samples in 0.3M hydrochloric acid for 7 days using the weight loss method. Microstructural examination of the samples before and after the corrosion test was conducted using a metallurgical optical microscope. The results obtained indicated that as the degree of deformation increased, the corrosion resistance of the cold-rolled samples decreased. Stress relief annealing improved the corrosion resistance of the cold-rolled low-carbon steel sample. The microstructure obtained revealed that the low-carbon steel is majorly composed of ferrite with some patches of pearlite.

Keywords: Acidic medium, annealing, cold rolling, corrosion penetration rate, hydrochloric acid, low-carbon steel

1. INTRODUCTION

Corrosion is an electrochemical, destructive and unintentional attack of a metal which is crucial to the safety and economy. Contamination of food and drugs due to corrosion of metallic components of agricultural and pharmaceutical plants can affect human health or lead to loss of lives. It has been estimated that approximately 5% of an industrialized nation's income is spent on corrosion prevention and the maintenance or replacement of products lost or destroyed due to corrosion reactions (Callister and Rethwisch, 2018; Oke et al., 2018). The corrosion penetration rate (CPR) is an important parameter that defines the rate of material removal as a consequence of the chemical action, it is the thickness loss of material per unit of time. For most applications, a corrosion penetration rate of less than about 20 mpy (0.50 mm/yr) is acceptable. The type of environment to which a material is exposed is a major factor in determining the type and rate of corrosion. Most industrial environments are rich in moisture, organic solvents, elemental gases, inorganic salts, bases and acidic solutions, all of which promote corrosion. The exposure of metals to these corrosive environments can, however, lead to sudden failure during service conditions. The soils in which underground structures like pipelines are buried are usually

corrosive due to bacterial action and the presence of moisture, oxygen, acids and organic solvents. However, plain carbon steels are a common candidate for these underground structures due to their lower cost (Atadious et al., 2021; Callister and Rethwisch, 2018).

Steel is one of the most widely used ferrous metals in the world. It is majorly composed of iron (90–99%) and carbon with the possible addition of some alloying elements. The percentage of carbon in most steel varies from above 0% to approximately 1% (Atadious et al., 2021; Brandt et al., 2005). There are many ways to classify steel but the most widely used classification is based on chemical composition. Steel is classified either as carbon steel or alloy steel. The carbon steel also called plain carbon steel is the most common type of steel and 90% of all steel made is carbon steel. This type of steel has few alloying elements and it is cheaper than alloy steel which contains more alloying elements responsible for the unique qualities they offer (ASM International, 1990). Carbon steels are further classified based on the percentage of carbon as low-carbon, medium-carbon and high-carbon steel. Low-carbon steel also known as mild steel contains less than 0.30% wt carbon, medium-carbon steel contains between 0.30 and 0.60% wt carbon while high-carbon steel has more than 0.60% wt carbon. Variation in the percentage of carbon content is responsible for the different mechanical properties such as strength, ductility, hardness, etc. Although low-carbon steel has relatively limited corrosion resistance, it is a preferred candidate in marine applications, chemical operations, petroleum production, refineries, pipeline, transportation, mining construction and metal processing equipment due to its low cost, good formability, weldability and strength (Ayodele and Nenuwa, 2013; Dwivedi et al., 2017; Readon, 2011).

Cold rolling is a common manufacturing process in which metal is deformed by passing it through rollers at a temperature below its recrystallization temperature. It is used to shape metal to the required dimension and improve its mechanical properties. Cold rolling increases the yield strength and hardness of metal with a decrease in ductility by introducing defects into the metal's crystal structure. These defects prevent further slip and can reduce the grain size of the metal, resulting in Hall-Petch hardening. Cold rolling is often used to decrease the thickness of the plate and sheet metal. The advantages of cold working over hot working include a higher quality surface finish, better mechanical properties and closer dimensional control of the finished piece. However, cold-rolled carbon steel is more susceptible to corrosion than the same material in its annealed state (Callister and Rethwisch, 2018; Uzorh, 2013).

Low-carbon steels are usually exposed to the action of acids in the industries during processes such as acid pickling, industrial acid cleaning, cleaning of oil refinery equipment, oil well acidizing and acid descaling. Hydrochloric acid is the least expensive and most widely used in industrial cleaning, petroleum processes and oil well acidification. The continuous exposure of low-carbon steel to this corrosive medium can lead to sudden failure of the material in service (ASM International, 2006; Jafar et al., 2022; Osarolube et al., 2008). Therefore, it is imperative to examine the corrosion behaviour of low-carbon steel when exposed to an acidic environment because this is an important factor in the selection of appropriate materials that can withstand such aggressive service conditions. The focus of this research is to investigate the corrosion rate of low-carbon steel in a hydrochloric acid environment.

2. MATERIAL AND EXPERIMENTAL METHODS

Material selection and sample preparation

A cylindrical low-carbon steel rod with a 12mm diameter was obtained from a commercial steel shop. Spectrometric analysis was carried out on the steel sample with the aid of a Spark analyzer and the result (Table 1). The result showed that the steel has a carbon content of 0.1122% which confirmed. The 12mm cylindrical rod was machined on a lathe machine to a 9mm diameter rod and the machined steel rod was then cut into eleven pieces of length 23mm. One of these samples was not further processed and it served as the reference sample while five of the samples were cold-rolled at 10%, 20%, 30%, 40% and 50% deformation. The other five samples were similarly cold-rolled and annealed inside a furnace at 700°C, they were held there for one hour and finally cooled in the furnace to room temperature. These annealed samples were polished to remove the oxide scale on them.

Table 1 Spectrometric analysis of the steel

| Element | C | Si | S | P | Mn | Ni | Cr | Mo | V | Cu |
|---------|--------|--------|--------|--------|--------|--------|--------|--------|---------|--------|
| % Comp. | 0.1122 | 0.1560 | 0.0479 | 0.0369 | 0.6102 | 0.1072 | 0.0864 | 0.0129 | 0.0018 | 0.3218 |
| Element | W | As | Sn | Co | Al | Pb | Ca | Zn | Fe | |
| % Comp. | 0.0011 | 0.0038 | 0.0388 | 0.0103 | 0.0000 | 0.0002 | 0.0000 | 0.0048 | 98.4477 | |

Corrosion test

Eleven similar corrosion media was prepared by pouring 100ml of 0.3M hydrochloric acid (HCl) solution inside a conical flask. All eleven prepared steel samples were initially weighed using Ohaus portable advanced digital scale. Each sample was inserted inside

the corrosion medium to obtain a total of eleven experimental set-ups. The weight of the corroded steel samples was taken at a regular interval of 24 hours for 7 days for each set-up. The weight losses for all the samples were calculated and recorded. The corrosion penetration rate was then calculated using Equation 1 (Callister and Rethwisch, 2018; Fontana, 1987).

$$\text{Corrosion penetration rate (CPR) in mm/yr} = \frac{KW}{\rho At} \quad (1)$$

W = Weight loss (g)

K = 87600

ρ = Density of low carbon steel (g/cm^3) = 7.85 g/cm^3 (Callister and Rethwisch, 2018)

A = Total surface area of the steel sample (cm^2)

t = Time of exposure (hrs.)

Microstructural examination

The specimens for microstructural examination were prepared by the conventional metallographic preparation method of grinding and polishing the samples on rotating polishing cloths to obtain a mirror-like surface. The polished surface was then etched with Nital, the surfaces were later cleaned and the microstructures were observed under the metallurgical optical microscope.

3. RESULTS AND DISCUSSION

Corrosion behaviour of the low carbon steel

Figure 1 presented the graph of corrosion rate against exposure time for the cold rolled low carbon steel in HCl. From the graph, the steel generally exhibited an increase in corrosion rate with a higher percentage of deformation, the sample cold rolled at 10% reduction had the lowest corrosion rate of between 53.06mm/y and 50.85mm/y while the sample cold rolled at 50% reduction had the highest corrosion rate of between 119.38mm/y and 79.58mm/y. Samples A3 and A4 which were deformed at 30% and 40% respectively had almost similar corrosion behaviour. The as-received sample, AC had the overall lowest corrosion rate ranging between 41.78mm/y and 35.81mm/y. This trend implies that as the degree of deformation increases, the corrosion resistance decreases. However, it was noted that the value of the corrosion rate after 24 hours was higher than its value after 144 hours, there was a gradual decline in the corrosion rate as the time of exposure increased. This could be attributed to the formation of a protective film on the metal surface which will retard the corrosion rate.

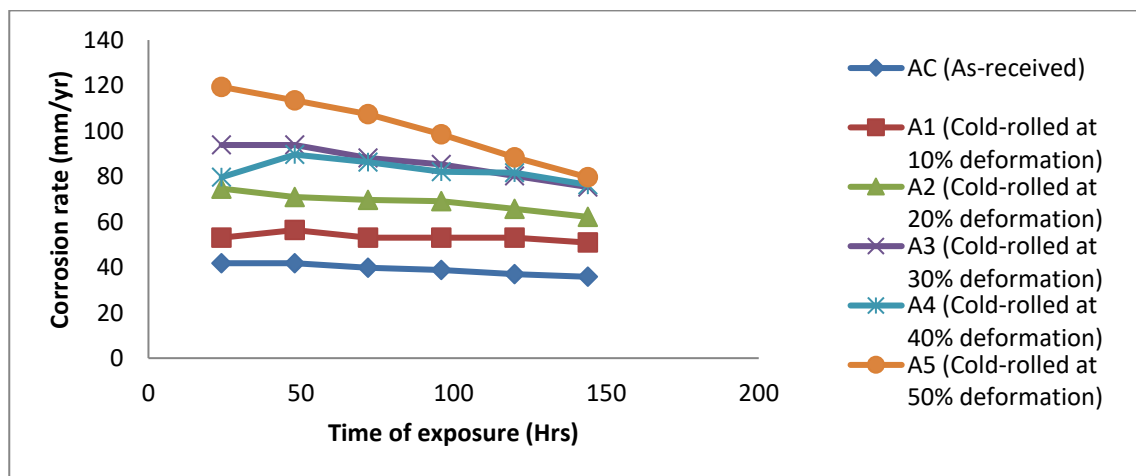


Figure 1 Corrosion rate (mm/yr) against the time of exposure (hrs) for cold-rolled steel in HCl

Figure 2 is the graph of the corrosion rate against the time of exposure for the cold-rolled and annealed low-carbon steel in HCl. From the graph, sample A5A cold rolled at 50% reduction had the highest corrosion rate of 83.56mm/y – 63.67mm/y while samples A3A and A4A had almost the same corrosion behaviour. A1A had a lower corrosion rate of between 53.06mm/y and 50.85mm/y while the corrosion rate of A2A is slightly higher than that of A1A. Sample AC which is the as-received (control) sample had the overall lowest corrosion rate. This also implies that as the percentage of deformation increases the corrosion resistance reduces. There was also a general improvement in the corrosion resistance as the time of exposure increased.

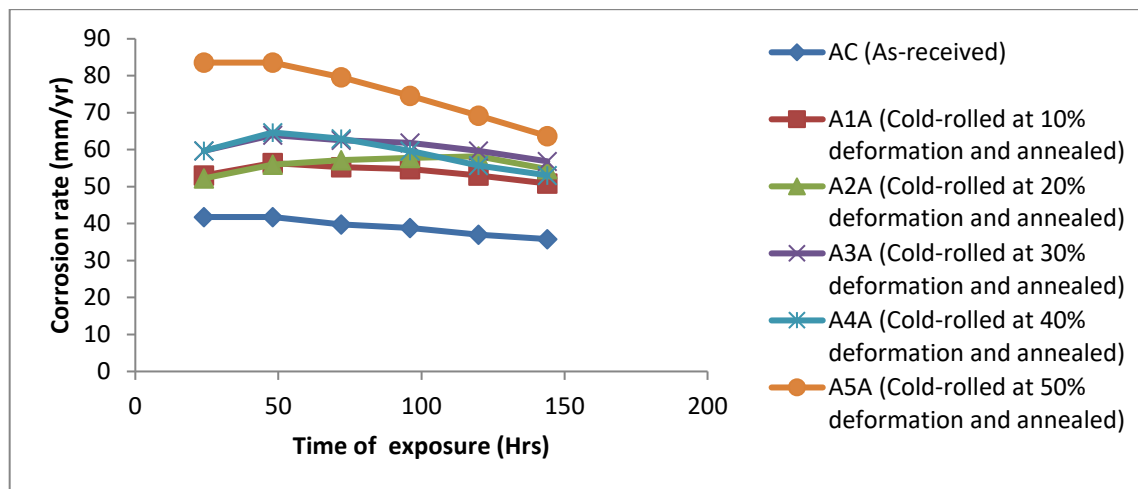


Figure 2 Corrosion rate (mm/yr) against the time of exposure (hrs) for cold-rolled and annealed steel in HCl

Figure 3 is the graph of the average corrosion rate against percentage deformation for the low-carbon steel. Curve A which represents the plot for the steel that was only cold rolled had the overall highest average corrosion rate with respect to the degree of deformation ranging from 39.16 to 101.11 mm/y. On annealing the cold rolled steel samples, the corrosion rate declined as shown by curve AA with values ranging from 39.16 and 75.7 mm/y. The corrosion rate increases gradually with an increase in percentage deformation and there was a decline at 40% deformation. These results further confirmed that the corrosion resistance of low-carbon steel reduces as the percentage deformation increases. Moreover, on annealing low-carbon steel samples which were earlier cold rolled, the corrosion resistance of the steel was improved.

From the results in Figure 1 and 2, it was observed that the as-received low carbon steel samples exhibited a lower corrosion rate than the other samples that were cold rolled and also annealed hence, having a higher corrosion resistance than other samples. This confirms that cold-working low-carbon steel minimizes its corrosion resistance and as the percentage deformation of the low-carbon steel increases the corrosion rate also increases. The higher corrosion rate of cold-rolled low-carbon steel is can be attributed to the additional energy induced into the steel causing it to be thermodynamically unstable and more vulnerable to electrochemical reactions leading to corrosion (Kurc et al., 2010). Stress-relief annealing effectively improved the corrosion resistance of the cold-rolled low-carbon steel as it was able to dissipate the residual stress earlier induced by the deformation process.

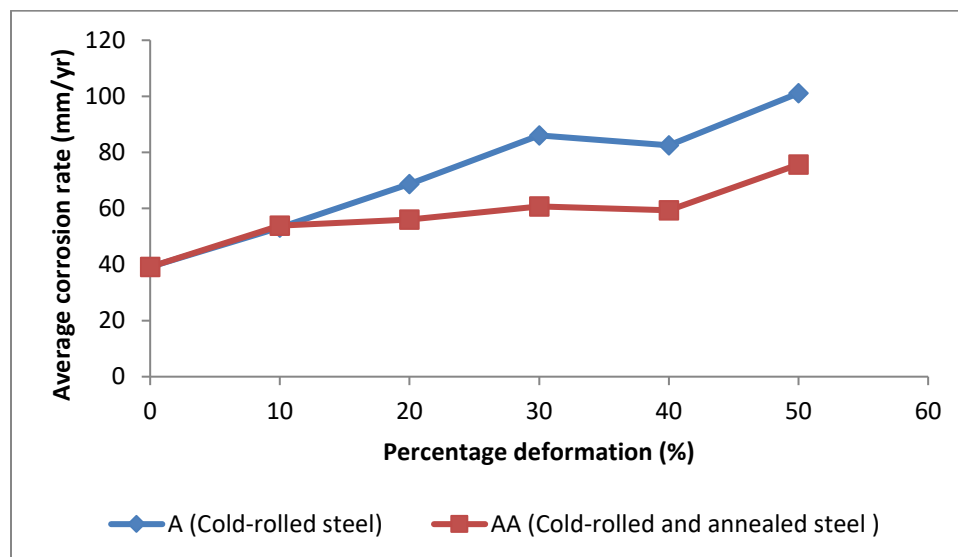


Figure 3 Average corrosion rate (mm/yr) against percentage deformation (%) for the steel

Micrographs of the samples

Figure 4, 5, 6, 7, 8, 9 and 10 are the microstructures as observed under the metallurgical optical microscope for the various steel samples. The samples subjected to 40% and 50% cold rolling were seriously corroded and they could not be polished for

microstructural examination. Figure 4a is the micrograph for the as-received low-carbon (LC) steel. The sparsely dispersed dark spots on the micrograph are the pearlite (which is a combination of ferrite and cementite) while the whitish regions are the ferrite. Ferrite which is more dominant in the matrix is very low in carbon and is responsible for the ductility of low-carbon steel. Figure 4b is the micrograph of the corroded low-carbon steel, there is no significant difference in the microstructure before and after corrosion except for corrosion along the grain boundaries which can only be visible under a more electron microscope. Figure 5, 7 and 9 presented the micrographs of the cold-rolled steels before and after the corrosion test which revealed microstructural grains aligned along the cold rolling direction. Figure 6, 8 and 10 are the micrographs of the cold-rolled and annealed low-carbon steel before and after corrosion, there was a re-crystallization of the grains due to the annealing process. This grain re-arrangement is responsible for the improvement in the corrosion resistance of samples that were cold-rolled and annealed compared to those that were only cold-rolled.

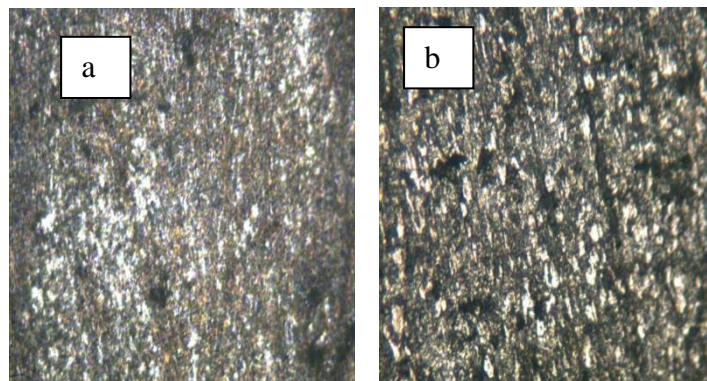


Figure 4 (a) As-received steel, LC (before corrosion) (b) LC (after corrosion)

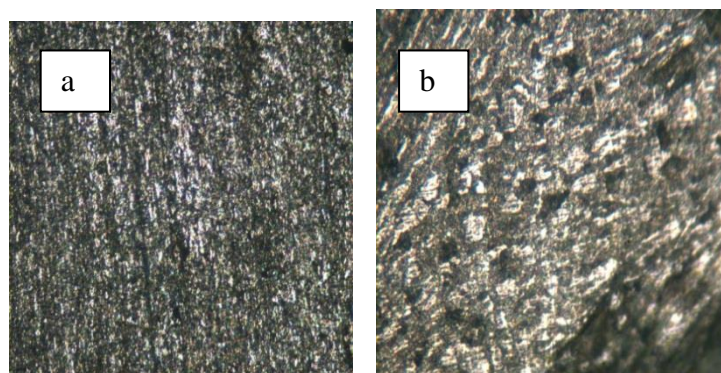


Figure 5 (a) 10% cold-rolled sample, L1 (before corrosion) (b) L1 (after corrosion)

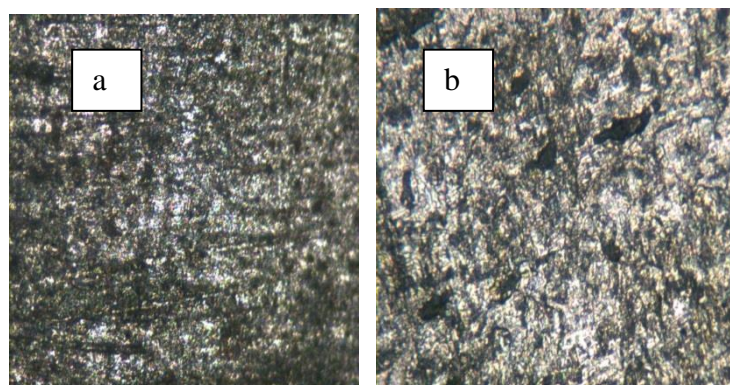


Figure 6 (a) 10% cold-rolled and annealed sample, L1A (before corrosion) (b) L1A (after corrosion)

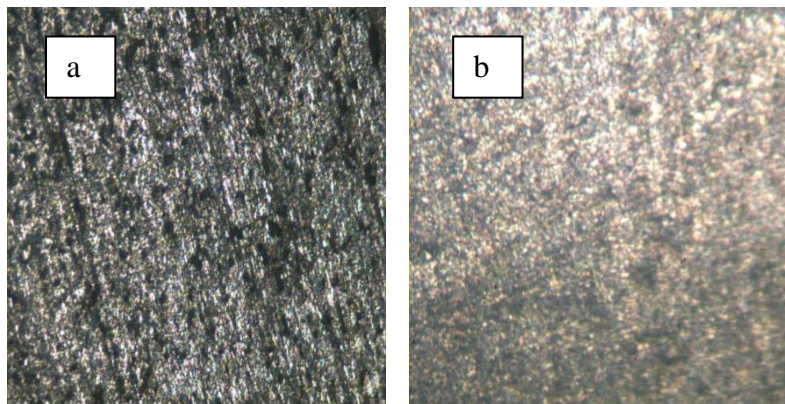


Figure 7 (a) 20% cold-rolled sample, L2 (before corrosion) (b) L2 (after corrosion)

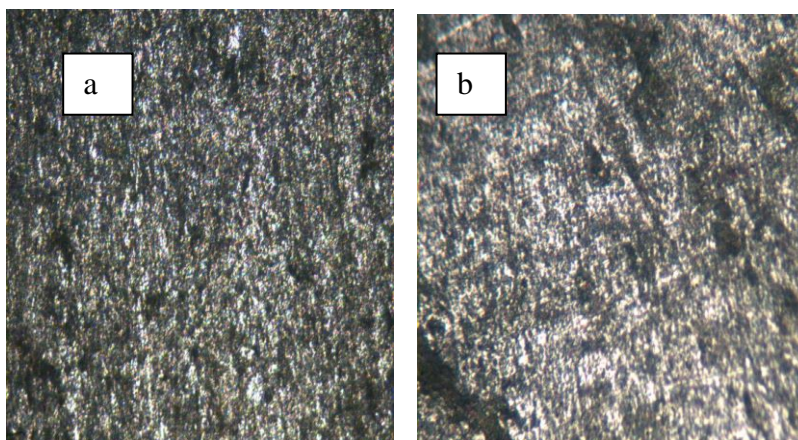


Figure 8 (a) 20% cold-rolled and annealed sample, L2A (before corrosion) (b) L2A (after corrosion)

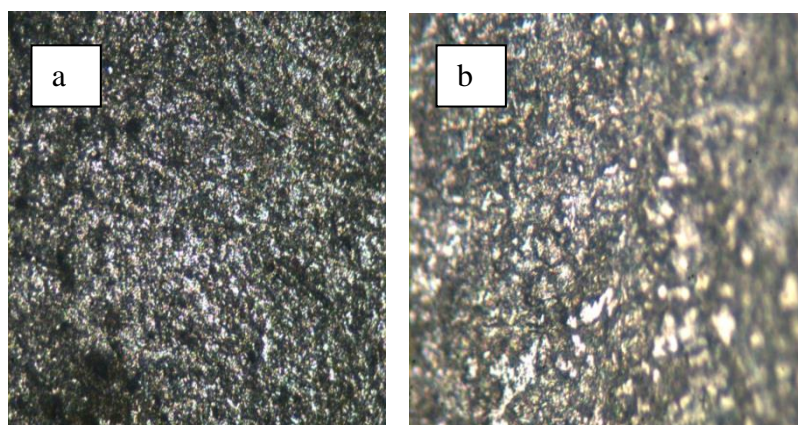


Figure 9 (a) 30% cold-rolled sample, L3 (before corrosion) (b) L3 (after corrosion)

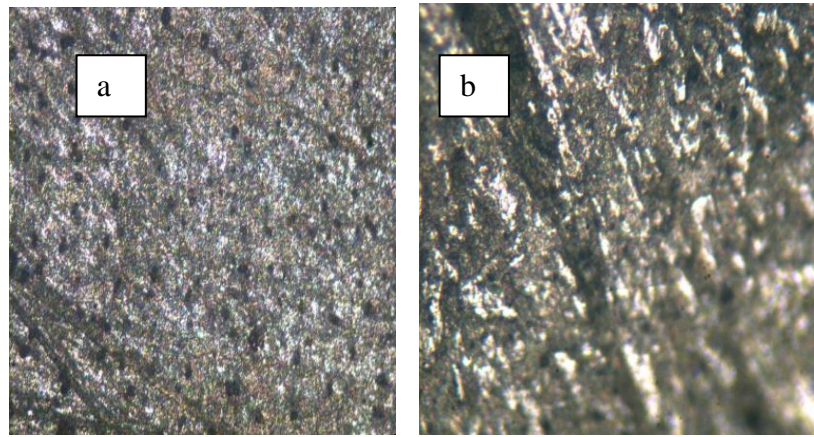


Figure 10 (a) 30% cold-rolled and annealed sample, L3A (before corrosion) (b) L3A (after corrosion)

4. CONCLUSIONS

From the result obtained in this study, it can be concluded that there was a general decrease in corrosion resistance as the percentage of deformation increased. It is, however, evident from this work that the samples cold-rolled and also annealed had lower corrosion rates and hence, better corrosion resistance than the samples that were only cold-rolled. Stress-relief annealing is effective in improving the corrosion resistance of cold-rolled low-carbon steel in aggressive media. The micrographs revealed that the low-carbon steel is majorly composed of ferrite with some patches of pearlite. Further work however needs to be carried out on the corrosion behaviour of cold-rolled low-carbon steel in other aggressive media.

Authorship contribution statement

Nenuwa OB: Conceptualization, validation, resources, investigation, methodology and writing of original draft

Oke OO: Investigation, validation, methodology, review and editing of manuscript

Informed consent

Not applicable.

Ethical approval

Not applicable.

Conflicts of interests

The authors declare that there are no conflicts of interests.

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Data and materials availability

All data associated with this study are present in the paper.

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